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9-Benzylidene-2,7-dichloro-4-oxiranyl-9*H*-fluorene

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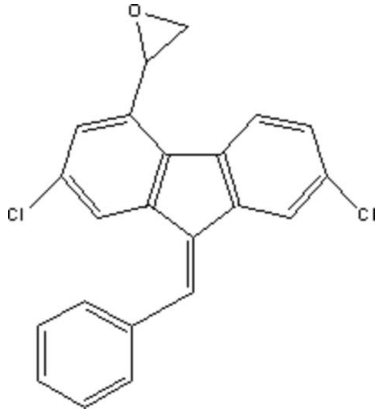
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.049; wR factor = 0.148; data-to-parameter ratio = 11.6.

The molecule of the title compound, $C_{22}H_{14}Cl_2O$, is built up from three fused rings, two six-membered and one five-membered, which are coplanar to within 0.015 (3) Å. The dihedral angles between the fluorene ring plane and the benzene and oxirane rings are 58.83 (13) and 55.99 (34)°, respectively. C—H···O hydrogen bonds link the molecules into sheets running parallel to the (010) plane. The crystal studied was an inversion twin.

Related literature

For related literature, see: Deng *et al.* (2000); Hamilton *et al.* (1995); Perry *et al.* (1999); Rao & Hu (2005, 2006); Alcaro *et al.* (2005).



Experimental

Crystal data

$C_{22}H_{14}Cl_2O$
M_r = 365.23
Monoclinic, *Pc*
a = 4.4860 (15) Å
b = 20.379 (7) Å
c = 9.497 (3) Å
β = 94.651 (4)°
V = 865.4 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 298 (2) K
0.20 × 0.15 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: multi-scan (Higashi, 1995)
T_{min} = 0.929, T_{max} = 0.960
4306 measured reflections
2638 independent reflections
2124 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.148
S = 1.05
2638 reflections
227 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), with 700 Friedel pairs
Flack parameter: 0.35 (11)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^{i}	0.93	2.49	3.388 (6)	163
C22—H22A···O1^{ii}	0.97	2.51	3.140 (7)	122

Symmetry codes: (i) $x+1, y, z+1$; (ii) $x+1, y, z$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2271).

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supplementary materials

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9-Benzylidene-2,7-dichloro-4-oxiranyl-9*H*-fluorene

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Comment

Fluorene derivatives have a high potential for biological activity (Stefano *et al.*, 2005; Perry *et al.*, 1999; Hamilton *et al.*, 1995). In a continuation of our work on the structure-activity relationships (Rao *et al.*, 2005, 2006), we have obtained a pale yellow crystalline compound (I) as the product of the reaction of benzaldehyde and 2-(2,7-dichloro-9*H*-fluoren-4-yl)oxirane.

The molecular structure of (I) is built up from three fused rings, two of which are six-membered and one five-membered (Fig. 1). The three rings in the fluorene are coplanar within −0.0145 (34) Å. The dihedral angles between the fluorene ring plane and the C15—C20 and C21/C22/O2 planes are 58.83 (13) and 55.99 (34) ^{o}, respectively.

The most interesting feature of the structure of (I) is the occurrence of C—H···O hydrogen bonds linking the molecules into sheets running parallel to the (0 1 0) plane·(Table 1, Fig. 2).

Experimental

The title compound was prepared from benzaldehyde and 2-(2,7-dichloro-9*H*-fluoren-4-yl)oxirane, according to the procedure of Deng *et al.* (2000). A solution of the compound in ethanol was concentrated gradually at room temperature to afford pale yellow prisms.

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.97 Å (methylene) or 0.98 Å (methine) with U_{iso}(H) = 1.2U_{eq}(C).

The value of the Flack parameter determined by using a twin refinement indicates the occurrence of a twin by inversion with two domains in the ratio 0.35/0.65.

Figures

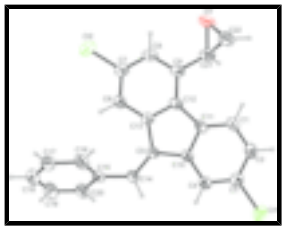

Fig. 1. The structure of (I), showing the atom-labelling scheme. Ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

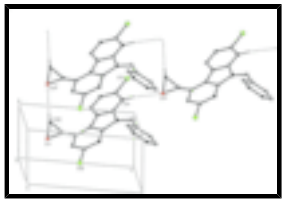

Fig. 2. Partial packing view showing the C—H···O hydrogen bonding interaction. The H bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. (i) $x + 1, y, z + 1$. (ii) $x + 1, y, z$.]

9-Benzylidene-2,7-dichloro-4-oxiranyl-9*H*-fluorene

Crystal data

$C_{22}H_{14}Cl_2O$	$F_{000} = 376$
$M_r = 365.23$	$D_x = 1.402$ Mg m^{-3}
Monoclinic, *Pc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P -2yc	Cell parameters from 994 reflections
a = 4.4860 (15) Å	θ = 2.4–23.8º
b = 20.379 (7) Å	μ = 0.38 mm^{-1}
c = 9.497 (3) Å	*T* = 298 (2) K
β = 94.651 (4)º	Prismatic, pale yellow
V = 865.4 (5) Å^3	0.20 × 0.15 × 0.12 mm
Z = 2	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.038$
Radiation source: fine-focus sealed tube	$\theta_{max} = 27.2$º
Monochromator: graphite	$\theta_{min} = 1.0$º
T = 298(2) K	*h* = −4→5
ω/2θ scans	*k* = −26→24
Absorption correction: multi-scan (Higashi, 1995)	*l* = −10→12
$T_{min} = 0.929$, $T_{max} = 0.960$	3 standard reflections
4306 measured reflections	every 60 min
2638 independent reflections	intensity decay: 0.3%
2124 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0919P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.148$	$(\Delta/\sigma)_{max} < 0.001$
S = 1.05	$\Delta\rho_{max} = 0.32$ e Å^{-3}
2638 reflections	$\Delta\rho_{min} = -0.27$ e Å^{-3}

227 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Extinction correction: none

Absolute structure: Flack parameter (Flack, 1983) using 700 Friedel pairs

Flack parameter: 0.35 (11)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	1.6375 (3)	0.03547 (6)	0.93615 (15)	0.0679 (4)
Cl2	0.1836 (3)	0.38150 (6)	0.50229 (17)	0.0736 (4)
O1	0.4761 (8)	0.1670 (2)	0.2348 (4)	0.0819 (12)
C1	1.0708 (10)	0.0977 (2)	0.6175 (5)	0.0514 (10)
H1	0.9998	0.0831	0.5283	0.062*
C2	1.2736 (11)	0.0604 (2)	0.7012 (5)	0.0544 (11)
H2	1.3397	0.0204	0.6682	0.065*
C3	1.3775 (10)	0.0826 (2)	0.8335 (5)	0.0512 (10)
C4	1.2874 (10)	0.14140 (19)	0.8864 (5)	0.0467 (9)
H4	1.3611	0.1560	0.9753	0.056*
C5	0.9496 (9)	0.24314 (18)	0.8327 (4)	0.0413 (8)
C6	0.5686 (10)	0.31227 (19)	0.6740 (5)	0.0477 (9)
H6	0.5518	0.3458	0.7393	0.057*
C7	0.4102 (10)	0.3140 (2)	0.5436 (5)	0.0520 (10)
C8	0.4260 (10)	0.2641 (2)	0.4468 (5)	0.0526 (10)
H8	0.3163	0.2671	0.3596	0.063*
C9	0.6010 (9)	0.2101 (2)	0.4770 (4)	0.0478 (9)
C10	1.0833 (9)	0.17822 (18)	0.8030 (4)	0.0399 (8)
C11	0.9758 (9)	0.15681 (18)	0.6685 (4)	0.0432 (9)
C12	0.7706 (9)	0.20730 (19)	0.6073 (4)	0.0437 (9)
C13	0.7543 (9)	0.25875 (19)	0.7048 (4)	0.0427 (8)
C14	0.9987 (10)	0.27523 (18)	0.9559 (5)	0.0496 (10)
H14	1.1155	0.2530	1.0259	0.059*
C15	0.8944 (10)	0.3404 (2)	0.9959 (5)	0.0526 (10)
C16	0.9433 (14)	0.3953 (2)	0.9159 (6)	0.0716 (15)
H16	1.0384	0.3912	0.8329	0.086*
C17	0.8518 (17)	0.4565 (2)	0.9586 (7)	0.0823 (18)

H17	0.8941	0.4935	0.9065	0.099*
C18	0.7000 (16)	0.4630 (3)	1.0767 (8)	0.091 (2)
H18	0.6306	0.5039	1.1025	0.109*
C19	0.6499 (17)	0.4083 (3)	1.1577 (7)	0.091 (2)
H19	0.5496	0.4125	1.2391	0.110*
C20	0.7493 (13)	0.3471 (2)	1.1173 (5)	0.0694 (14)
H20	0.7178	0.3105	1.1727	0.083*
C21	0.6020 (11)	0.1543 (2)	0.3753 (5)	0.0617 (12)
H21	0.5518	0.1115	0.4138	0.074*
C22	0.7848 (13)	0.1521 (3)	0.2568 (6)	0.0767 (16)
H22A	0.9263	0.1875	0.2473	0.092*
H22B	0.8506	0.1094	0.2263	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0768 (8)	0.0586 (6)	0.0689 (8)	0.0160 (6)	0.0109 (6)	0.0178 (5)
Cl2	0.0666 (8)	0.0730 (8)	0.0797 (9)	0.0120 (6)	−0.0031 (6)	0.0193 (7)
O1	0.064 (2)	0.134 (3)	0.0453 (19)	0.012 (2)	−0.0138 (16)	−0.024 (2)
C1	0.062 (3)	0.050 (2)	0.043 (2)	−0.010 (2)	0.010 (2)	−0.0101 (18)
C2	0.061 (3)	0.044 (2)	0.060 (3)	−0.004 (2)	0.019 (2)	−0.004 (2)
C3	0.052 (3)	0.052 (2)	0.051 (3)	0.0008 (19)	0.016 (2)	0.0114 (19)
C4	0.058 (2)	0.0418 (19)	0.040 (2)	−0.0040 (18)	0.0078 (18)	0.0021 (16)
C5	0.049 (2)	0.0397 (19)	0.0354 (19)	−0.0034 (16)	0.0036 (16)	−0.0020 (15)
C6	0.050 (2)	0.047 (2)	0.047 (2)	−0.0060 (18)	0.0040 (18)	−0.0036 (18)
C7	0.046 (2)	0.055 (2)	0.055 (3)	−0.0049 (19)	0.0009 (19)	0.010 (2)
C8	0.051 (3)	0.066 (3)	0.040 (2)	−0.010 (2)	−0.0039 (18)	0.006 (2)
C9	0.048 (2)	0.060 (2)	0.035 (2)	−0.0121 (18)	0.0026 (17)	0.0007 (17)
C10	0.048 (2)	0.0397 (18)	0.0325 (19)	−0.0085 (16)	0.0063 (16)	0.0007 (15)
C11	0.046 (2)	0.049 (2)	0.036 (2)	−0.0116 (18)	0.0110 (17)	−0.0061 (17)
C12	0.044 (2)	0.052 (2)	0.036 (2)	−0.0101 (17)	0.0045 (16)	−0.0004 (17)
C13	0.041 (2)	0.049 (2)	0.039 (2)	−0.0075 (16)	0.0039 (15)	0.0030 (16)
C14	0.064 (3)	0.045 (2)	0.039 (2)	−0.0004 (19)	0.0006 (18)	−0.0059 (17)
C15	0.063 (3)	0.052 (2)	0.041 (2)	−0.0025 (19)	−0.0054 (19)	−0.0112 (17)
C16	0.099 (4)	0.055 (3)	0.059 (3)	−0.013 (3)	−0.002 (3)	−0.012 (2)
C17	0.122 (5)	0.046 (2)	0.074 (4)	−0.002 (3)	−0.027 (4)	−0.003 (2)
C18	0.113 (6)	0.061 (3)	0.093 (5)	0.020 (3)	−0.015 (4)	−0.029 (3)
C19	0.115 (5)	0.089 (4)	0.072 (4)	0.024 (4)	0.014 (4)	−0.026 (3)
C20	0.093 (4)	0.065 (3)	0.051 (3)	0.003 (3)	0.009 (3)	−0.008 (2)
C21	0.064 (3)	0.071 (3)	0.048 (3)	−0.009 (2)	−0.011 (2)	−0.001 (2)
C22	0.073 (4)	0.105 (4)	0.052 (3)	0.006 (3)	0.004 (2)	−0.015 (3)

Geometric parameters (Å, °)

Cl1—C3	1.746 (5)	C9—C21	1.492 (6)
Cl2—C7	1.736 (4)	C10—C11	1.398 (6)
O1—C22	1.416 (7)	C11—C12	1.469 (6)
O1—C21	1.430 (6)	C12—C13	1.404 (6)
C1—C11	1.378 (6)	C14—C15	1.468 (6)

C1—C2	1.386 (7)	C14—H14	0.9300
C1—H1	0.9300	C15—C20	1.377 (7)
C2—C3	1.381 (7)	C15—C16	1.381 (7)
C2—H2	0.9300	C16—C17	1.384 (7)
C3—C4	1.372 (6)	C16—H16	0.9300
C4—C10	1.382 (6)	C17—C18	1.365 (10)
C4—H4	0.9300	C17—H17	0.9300
C5—C14	1.343 (5)	C18—C19	1.382 (9)
C5—C13	1.474 (6)	C18—H18	0.9300
C5—C10	1.489 (5)	C19—C20	1.389 (7)
C6—C7	1.377 (6)	C19—H19	0.9300
C6—C13	1.389 (6)	C20—H20	0.9300
C6—H6	0.9300	C21—C22	1.446 (7)
C7—C8	1.377 (6)	C21—H21	0.9800
C8—C9	1.369 (6)	C22—H22A	0.9700
C8—H8	0.9300	C22—H22B	0.9700
C9—C12	1.401 (6)		
C22—O1—C21	61.1 (3)	C6—C13—C12	120.7 (4)
C11—C1—C2	119.1 (4)	C6—C13—C5	130.5 (4)
C11—C1—H1	120.5	C12—C13—C5	108.8 (3)
C2—C1—H1	120.5	C5—C14—C15	129.2 (4)
C3—C2—C1	119.9 (4)	C5—C14—H14	115.4
C3—C2—H2	120.0	C15—C14—H14	115.4
C1—C2—H2	120.0	C20—C15—C16	119.2 (4)
C4—C3—C2	122.1 (4)	C20—C15—C14	119.3 (4)
C4—C3—Cl1	118.6 (4)	C16—C15—C14	121.6 (4)
C2—C3—Cl1	119.2 (3)	C15—C16—C17	120.4 (6)
C3—C4—C10	117.7 (4)	C15—C16—H16	119.8
C3—C4—H4	121.1	C17—C16—H16	119.8
C10—C4—H4	121.1	C18—C17—C16	120.5 (6)
C14—C5—C13	131.1 (4)	C18—C17—H17	119.8
C14—C5—C10	123.9 (4)	C16—C17—H17	119.8
C13—C5—C10	104.9 (3)	C17—C18—C19	119.6 (5)
C7—C6—C13	117.8 (4)	C17—C18—H18	120.2
C7—C6—H6	121.1	C19—C18—H18	120.2
C13—C6—H6	121.1	C18—C19—C20	120.0 (6)
C6—C7—C8	122.0 (4)	C18—C19—H19	120.0
C6—C7—Cl2	118.3 (4)	C20—C19—H19	120.0
C8—C7—Cl2	119.7 (4)	C15—C20—C19	120.3 (5)
C9—C8—C7	120.9 (4)	C15—C20—H20	119.8
C9—C8—H8	119.5	C19—C20—H20	119.8
C7—C8—H8	119.5	O1—C21—C22	59.0 (3)
C8—C9—C12	118.6 (4)	O1—C21—C9	116.4 (4)
C8—C9—C21	120.6 (4)	C22—C21—C9	124.0 (5)
C12—C9—C21	120.8 (4)	O1—C21—H21	115.1
C4—C10—C11	121.1 (4)	C22—C21—H21	115.1
C4—C10—C5	129.2 (4)	C9—C21—H21	115.1
C11—C10—C5	109.6 (3)	O1—C22—C21	59.9 (3)
C1—C11—C10	120.0 (4)	O1—C22—H22A	117.8

C1—C11—C12	132.5 (4)	C21—C22—H22A	117.8
C10—C11—C12	107.5 (3)	O1—C22—H22B	117.8
C9—C12—C13	119.9 (4)	C21—C22—H22B	117.8
C9—C12—C11	131.0 (4)	H22A—C22—H22B	114.9
C13—C12—C11	109.1 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.93	2.49	3.388 (6)	163
C22—H22A···O1^{ii}	0.97	2.51	3.140 (7)	122

Symmetry codes: (i) *x*+1, *y*, *z*+1; (ii) *x*+1, *y*, *z*.

Fig. 1

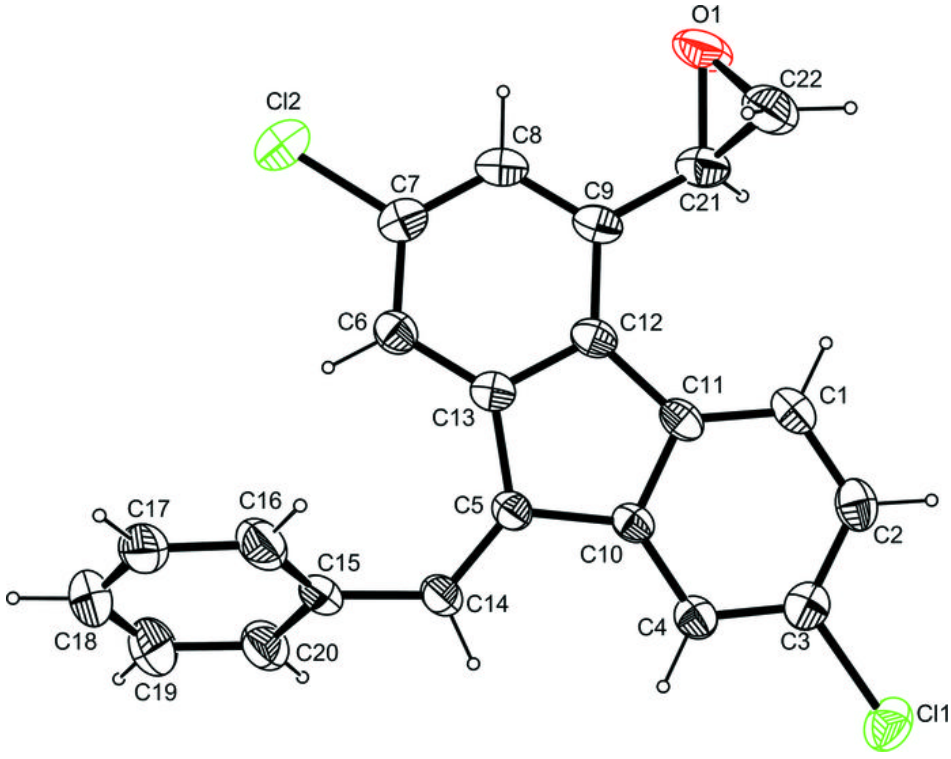
O1
C22
Cl2
C8
C7
C9
C21
C6
C12
C11
C1
C13
C5
C17
C16
C15
C10
C2
C14
C18
C20
C4
C3
C19
Cl1

Fig. 2